PTO: 2006-3924
Japanese Published Unexamined (Kokai) Patent Publication No. 59-223756; Publication Date: December 15, 1984; Application No. 58-099061; Application Date: June 2, 1983; Int. Cl.[3]: C09B 61/00; Inventor(s): Akira Yasuda et al.; Applicant: Sanei Chemical Industry Ltd.; Japanese Title: Antoshianin Shkiso no Seizouhouhou (Method for Production of Anthocyanin Pigment)

---

Specification

1. Title of Invention

Method for Production of Anthocyanin Pigment

2. Claim

A method for production of an anthocyanin pigment, characterized in that an acidic water extract of anthocyanin pigment containing foods such as purple corns, red cabbage, berries, grape berry skin and others is purified using cationic or absorbing resin; after the extract has been controlled to pH 7.0 or lower, it is treated with an ultrafiltration membrane (a 2000 to 200000 fractions).

3. Detailed Description of the Invention

This invention pertains to a pigment and aims to obtain a high purity anthocyanin pigment by an industrially advantageous means.

It is widely known that the anthocyanin pigment is contained in purple corns, red cabbage, grape berry skin, juices, berries and others. An obtaining of the anthocyanin pigment efficiently at a high quality from these raw materials becomes the issue of persons skilled in the art.

This invention is produced so as to correspond to the issue by obtaining a desired pigment at a high quality at a minimized loss at a low temperature range (40 or lower °C). The details are described hereinbelow.

First, a solution for an anthocyanin pigment is prepared. The anthocyanin pigment is stably eluded from the aforementioned raw materials in an acidic solution. As for acids used for the acidic solution, hydrochloric acid, sulfuric acid, other mineral acids, citric acid, tartaric acid, malic acid and other organic acids are used. The pH is preferably 4.0 or lower to improve the yield of the pigment. If the pH exceeds 4.0 to be the alkaline side, the yield decreases.

Cationic or absorbing resin is used for purifying an anthocyanin pigment containing solution obtained from an extraction process.

Deniolite XAD2, Deniolite XAD-4, Deniolite XAD-7, Diaion HP-50, Diaion HP-20 and others are used as absorbing resin. Duolite C-3, Duolite C-10, Duolite C-20, Duolite S-30 and others are used as cationic resin. The purifying method is as follows. By running the anthocyanin pigment containing acidic solution, the pigment is absorbed in the resin. After the resin has been rinsed with water (a fair stream or a back stream), the pigment is removed. It is important that the temperature is 40 or lower °C at this process. If the temperature exceeds 40°C, the yield of the pigment decreases to deteriorate the color phase.

Foreign substances are removed at the process, such as sugar, salts, fats and others. It is necessary to control the pH of the removed pigment. The reaching pH is in the range of 1.0 to 7.0. If the pH exceeds 7.0, the yield of the desired substance deteriorates.

At the next step, the purified solution is treated with an ultrafiltration film. The type of the film slightly varies by the raw material, and the molecular weight can generally be fractionated at 2000 to 200000 fractions (MW). The operation can be made by a regular ultrafiltration means. In the case of a red cabbage extract, the following conditions are optimal: a 5 $kg/cm^2$ pressure; a 15 $l/min/cm^2$ flow rate. Similar conditions are also applied to a purple corn extract. This solution is treated at a temperature of 40 or lower °C as similarly to as in the previous step. At this step, ash, protein, polysaccharides, acids and others are removed among foreign substances.

The obtained anthocyanin pigment is a desired anthocyanin pigment at a high quality.

According to the producing method for the invention, the anthocyanin pigment is at a high quality and can be obtained at a high yield. When the anthocyanin pigment is applied to various foods that require transparency, the foods increase the vividness. Turbidity, precipitation and other disadvantages are also eliminated.

Thereby, the invention completely achieves the purpose.

| | Red cabbage | | | Purple corns | Grape juice | Elder berries | |
|---|---|---|---|---|---|---|---|
| Treatment for resin | Duolite XAD-7 | Duolite XAD-7 | | Diaion HP-50 | Diaion HP-50 | Diaion HP-20 | |
| Ultrafiltration 1<br>Molecular weight cut off<br>Pressure $kg/cm^2$<br>Flow rate $l/min/cm^2$<br>Permeation flow rate $ml/m^2/hr$<br>Pigment yield % | [Please refer to the original description] | | | | | | |
| Ultrafiltration | [Please | | | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 2 Molecular weight cut off Pressure $kg/cm^2$ Flow rate $l/min/cm^2$ Permeation flow rate $ml/m^2/hr$ Pigment yield % Total yield Drink precipitate | refer to the original description] | | | | | | |

The permeation flow rate of the resin purified pigment solution does not decrease at the ultrafiltration. The pigment permeation at the second ultrafiltration is also high.

The pigment without having ultrafiltration generates a precipitate over time in use for drinking. When the pigment is used with other pigments and juices, a precipitate is generated depending on the presence or absence of electrostatic colloid. Such effects are not identified on the purified pigment of the invention.

Working Example 1

One part (weight; henceforth referred to as is) of purple corn seeds is immersed in 10 parts of a 1% (weight; henceforth referred to as is) sulfuric acid at 40°C for 30 hours so as to obtain red-purple anthocyanin pigment extract (10°C; henceforth referred to as is in the working example). After this solution has been absorbed in 20 parts of Diaion HP-50, a rinsing is applied with water. A pigment is then eluded using 2 parts of 58 volume % ethyl alcohol.

First, ultrafiltration using a membrane with a 6000 fractionation molecular weight (Nitto NTU2006) is applied to the pigment solution obtained after removing alcohol by a

distillation means so as to remove low molecular foreign substances such as ash, sulfuric acid and sugar. At the next step, after adjusting the pH to 3.0 using citric acid, low molecular foreign substances are removed, including ash, sulfuric acid and sugar. High molecular foreign substances are thereafter removed using a membrane (Nitto-NTU35100) with a 100000 fractionation molecular weight, including protein, starch and colloid. These ultrafiltration operations are performed at a 5 $kg/cm^2$ pressure at a 11 $l/min/cm^2$ flow rate. The permeation flow rates are 28 and 60 ($ml/m^2/hr$), respectively. Because of the pretreatment by resin, no clogging occurs during the ultrafiltration. Thereby, a higher permeation speed is obtained as well as an extremely high concentration level.

The obtained pigment demonstrates extremely transparent red. When the pigment is used for juice beverages, no precipitate occurs over time.

Working Example 2

After one part of pulverized red cabbage has been immersed in 20 parts of 1% citric acid water at 40°C for 30 hours, the mixture solution is filtered by a centrifugal means to obtain a red-purple anthocyanin pigment solution. This pigment solution is absorbed in one part of Duolite XAD-7. A rinsing is applied with water. A pigment is then eluded using one part of 58 v/v% ethyl alcohol. First, low molecular foreign substances are removed using a membrane (Nitto-NTU3508) with an 8000 fractionation molecular weight, including sugar, ash and acid. Ultrafiltration is then applied using a membrane (Nitto-NTU35100) with a 100000 fractionation molecular weight to remove

high molecular colloidal foreign substances including protein, starch and other colloidal substances.

When the obtained pigment is used for coloring carbonated beverages and juice beverages, no precipitate is identified over time. Since a red cabbage pigment contains a positive colloidal ion substance, when a pigment obtained by prior art method, if negative colloidal ions (a tannin substance) is presented in the juice, the negative colloidal ions are also precipitated. Juice beverages are colored transparent for the first time by the use of the pigment obtained by the method of the invention.

Working Example 3

After one part of dry elderly berries has been pulverized, they are immersed in 20 parts of 1% sulfuric acid at 40°C for 5 hours. The mixture is agitated. The filtered pigment solution is then absorbed in 20 parts of absorbing resin (Diaion HP-20). After rinsing with water, a pigment is eluded using one part of 58 v/v % ethyl alcohol. First, ultrafiltration is applied to the eluded pigment solution with alcohol removed by a distillation means at a 5000 fractionation molecular weight. Another ultrafiltration is thereafter applied at a 100000 fractionation molecular weight.

At these treatments, low molecular foreign substances including sulfuric acid and sugar and high molecular foreign substances including a tannin substance, protein and starch. The solution is transparent, and a precipitate hardly occurs over time.

U.S. Patent and Trademark Office
Translations Branch
4/12/06
Chisato Morohashi